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(54) Propylene polymer films and laminates.

(57) Disclosed is a film or sheet material comprising a blend of (1) a crystalline propylene polymer composition which is comprised of, by weight, (A) from about 30 to 65% of a copolymer of propylene with a C<sub>4</sub>-8 alpha-olefin containing from 80 to 98% propylene, and (B) 35-70% of a co- or terpolymer of propylene with ethylene and, optionally, a C<sub>4</sub>-8 alpha-olefin having an ethylene content of 5 to 10% when ethylene is the sole comonomer and an ethylene content of 0.5 to 5% and a C<sub>4</sub>-8 alpha-olefin content of 1 to 9.5% when both ethylene and a C<sub>4</sub>-8 alpha-olefin are used as comonomers, and (2) an olefin polymer.

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This invention relates to weldable thermoplastic films or sheets formed from blends of a propylene polymer composition with thermoplastic polymer materials.

In many film applications, such as packaging of foodstuffs, chemical and hazardous materials and in medical applications, the industry requires films having certain properties. In the packaging of foodstuffs, for example, the films must have high puncture resistance, high clarity and gloss, reduced permeability to gases and/or vapors and good weld strength. The films used to manufacture containers for chemicals and hazardous waste materials must have a high puncture resistance, high elongation strength, high tear resistance, chemical resistance and good weld strength. Films used in medical applications, such as blood bags, must have a high puncture resistance, low modulus, high tear resistance, autoclavability and good weld strength.

Films made from ethylene polymers, e.g., HDPE and LLDPE, and propylene polymers, such as crystalline homopolymers of propylene and random copolymers of propylene and ethylene do not provide such a combination of desirable properties.

A thermoplastic composition useful to form a film, which comprises olefin polymers characterized in that the mixture comprises

- (i) a copolymer of propylene with an alpha-olefin with 4 to 10 carbon atoms, having a content of propylene in the copolymer from 80 to 95 % by weight of the copolymer, and
- (ii) a second polymer of an alpha-olefin with 4 to 10 carbon atoms, copolymerized with an alpha-olefin with 2 to 10 carbon atoms which has a content from 0 to 10 % by weight of the copolymer,

is disclosed in FR-A-2-314 211.

A thermoplastic olefin resin composition composed of a blend of

- (i) 5 to 95 % by weight, based on the weight of the blend, of a random copolymer consisting essentially of 55 to 85 mole % of propylene and 45 to 15 mole % of 1-butene, and
- (ii) 5 to 95 % by weight, based on the weight of the blend, of an isotactic propylene resin containing 0 to 10 mole % of another alpha-olefin

is disclosed in US-A-4,211,852.

Both compositions are mechanically blended

Crystalline random copolymers of propylene and ethylene are frequently used to prepare such films because of the inherent clarity of such copolymers. However, films prepared from crystalline copolymers of propylene and less than 3% ethylene or blends thereof with other olefin polymers do not provide good seals or weld strength since the melting point of these copolymers or blends thereof is too close to the melting point of the base polymer layer they are laminated to or coextruded with, which is typically a crystalline homopolymer of propylene. Further the resulting laminate or coextruded material lacks dimensional integrity or stability.

When films are prepared from crystalline random copolymers of propylene and ethylene having about 5-6% ethylene, the melting point of said copolymer is decreased with respect to the melting point of the crystalline homopolymers of propylene so that such a copolymer film does have certain weldability properties. However, the insertion of the comonomer disrupts the crystalline structure with the concomitant formation of an increased fraction of xylene soluble polymer at room temperature. Consequently, the mechanical properties of films formed from such a copolymer are adversely affected. Furthermore, the presence of a high xylene soluble polymer fraction renders the film material susceptible to attack from organic substances and, therefore, unsuited for use in food packaging. Films prepared from blends of such crystalline copolymers of propylene with other polymers do not overcome these disadvantages.

Therefore, there is a need for a film or sheet material having a low seal initiation temperature as well as other desirable properties comprising a propylene polymer material having a low xylene soluble polymer fraction at room temperature and weldability.

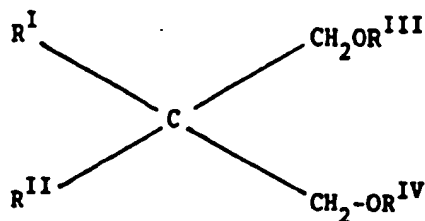
All parts and percentages used in this application are by weight unless otherwise specified. Ambient or room temperature is approximately 25 °C.

This invention provides a film or sheet material comprising a blend of

- (1) from about 5 to 45 % of a crystalline propylene polymer composition which is comprised of, by weight,
  - (i)
    - (A) from about 30 to 65 % of a copolymer consisting essentially of propylene with a C<sub>4-8</sub> alpha-olefin containing from 80 to 98 % propylene, and
    - (B) from about 35 to 70 % of a copolymer consisting essentially of propylene with ethylene having an ethylene content of 5 to 10 %;

or

- (ii)



where  $R^I$  and  $R^{II}$  are the same or different from each other, and are  $C_1-18$  straight or branched alkyl,  $C_5-18$  cycloalkyl or  $C_6-18$  aryl radicals;  $R^{III}$  and  $R^{IV}$  are the same or different, and are  $C_1-4$  straight or branched alkyl radicals. Typical ethers of this type and methods of preparing same are described in the U.S. application, Ser. No. 413,409, filed September 27, 1989, the disclosure of which is incorporated herein by reference. Examples of such ether compounds include 2-methyl-2-isopropyl-1,3-dimethoxypropane, 2,2-diisobutyl-1,3-dimethoxypropane and 2-isopropyl-2-cyclopentyl-1,3-dimethoxypropane.

The supported catalyst component can be prepared by milling a conventional anhydrous magnesium dihalide, i.e. an unactivated magnesium dihalide containing less than 1% water, the titanium compound and an electron donor compound under conditions which cause activation of the magnesium dihalide. The milled product is then treated one or more times with an excess of  $TiCl_4$  at a temperature from 90 and  $135^\circ C$  to washed repeatedly with a hydrocarbon (e.g. hexane) until all chlorine ions have disappeared from the wash.

Alternatively, the anhydrous magnesium halide is preactivated using known methods and then treated with an excess of  $TiCl_4$  containing an electron donor compound in solution at a temperature between 80 and  $135^\circ C$ . The treatment with  $TiCl_4$  is then repeated, and the solid is then washed with hexane or other suitable hydrocarbon solvent to eliminate all traces of unreacted  $TiCl_4$ . The magnesium dihalide compound or a complex thereof may be formed in situ from a magnesium compound capable of forming same upon treatment with a halide-containing titanium compound which is preferably  $TiCl_4$ .

In another method, the solid catalyst support may be prepared by forming an adduct, preferably in spherical particle form, of magnesium dichloride and an alcohol, such as ethanol, propanol, butanol, isobutanol and 2-ethyl-hexanol, wherein the molar ratio is 1:1 to 1:3, which then is treated with an excess of  $TiCl_4$  containing an electron donor in solution. The temperature ranges generally between 80 and  $120^\circ C$ . The solid is isolated and treated again with  $TiCl_4$ . The solid is separated and washed with a hydrocarbon until all chlorine ions have disappeared from the wash.

In yet another method, alkoxy magnesium compounds and alkoxy magnesium chloride compounds (the alkoxy magnesium chloride compounds being prepared according to the method described in U.S. Patent No. 4,220,554, the disclosure of said method being incorporated herein by reference), are treated with an excess of  $TiCl_4$  containing an electron donor compound in solution, under the reaction conditions described above.

In the solid catalyst component, the Ti compound, expressed as Ti, is generally present in an amount from 0.5 to 10% by weight; and the amount of electron donor compound fixed on the solid counterpart (inside donor) is generally from 5 to 20 mole % with respect to the magnesium dihalide. Useful titanium compounds for the preparation of the solid catalyst component are the halides and the alkoxy halides of titanium. Titanium tetrachloride is the preferred compound. Satisfactory results are obtained also with titanium trihalides and with alkoxy halides of titanium, such as  $TiCl_3OR$  where R is a phenyl radical.

In addition to the above reactions which result in the formation of magnesium dihalides in active form, there are other reactions known in the literature which result in the formation of magnesium dihalide in active form from magnesium compounds other than the halides, such as alkoxy magnesium compounds and magnesium carbonate.

The active form of the magnesium dihalide in the solid catalyst component is evidenced in the X-ray spectrum of the solid catalyst component by the absence of the high intensity diffraction line which appears in the X-ray spectrum of the unactivated magnesium dihalide (having a surface area less than  $3 \text{ m}^2/\text{g}$ ) and instead there is a halo with the maximum intensity shifted with respect to the position of the high intensity diffraction line of the unactivated magnesium dihalide, or by the fact that said high intensity diffraction line shows a broadening having a half peak breadth at least 30% greater than the one of the high intensity diffraction line of the unactivated magnesium dihalide. The most active forms are those in which the aforementioned halo appears in the X-ray spectrum of the solid catalyst component.

Table 1A

Examples	1	2
<b>FIRST GAS PHASE REACTOR</b>		
Temperature, °C	65	65
Pressure, atm.	17	17
Residence time, min.	75	75
H <sub>2</sub> /C <sub>2</sub> , mol %	0.003	0.035
H <sub>2</sub> /C <sub>2</sub> , mol %	0.151	-
C <sub>2</sub> /C <sub>2</sub> + C <sub>3</sub> , mol %	0.023	-
C <sub>4</sub> /C <sub>4</sub> + C <sub>3</sub> , mol %	0.044	0.178
<b>SECOND GAS PHASE REACTOR</b>		
Temperature, °C	70	70
Pressure, atm.	17	20
Residence time, min.	45	45
H <sub>2</sub> /C <sub>2</sub> , mol %	0.005	0.025
H <sub>2</sub> /C <sub>2</sub> , mol %	-	0.505
C <sub>2</sub> /C <sub>2</sub> + C <sub>3</sub> , mol %	-	0.041
C <sub>4</sub> /C <sub>4</sub> + C <sub>3</sub> , mol %	0.214	-

Table 1B

Examples	1	2
Fraction (A), wt %	52	45.1
Fraction (B), wt %	48	54.9
Ethylene in (B), wt. %	2.5	3.8
Butene-1 in (A), wt %	14.2	15.3
Butene-1 in (B), wt %	3.6	-
Melting point*	-	132.8
MIL*, g/10 min.	1.65	6.07
I.V.*, dl/g	2.31	1.69
Xylene solubles at 25°C, wt %*	15.72	11.72
I.V. of xylene solubles, dl/g	1.78	1.02
Hexene solubles at 50°C, wt %*	3	5
Yield g polymer		
g catalyst component	6,500	20,000

\* on the final propylene polymer composition

The seal initiation temperature (S.I.T.) of the propylene polymer compositions of examples of 1 and 2 is determined by extruding said compositions at about 200°C into films 50 microns thick. Each of the resulting films is laid on a 560 micron thick film layer of polypropylene having an isotacticity index of 97 and a melt index of 4.5 g/10 min. A plate press at 200°C and at a pressure of 9000 kg. is placed on top of the superimposed film layers for 5 minutes to form a laminate of the two film layers. The resulting laminate is stretched 6 times its height in the machine and cross directions using a TM Long film stretcher, thereby obtaining a laminated film about 20 microns thick. From these biaxially oriented laminated films, 5 x 10 cm test pieces are cut. Two of the above described test pieces for each composition are superimposed with the layers made of the propylene polymer composition of examples 1 or 2, as the case may be, facing each other. The superimposed films are then sealed along the 5 cm long side with a Sentinel Combination Laboratory sealer, Model 12-12AS. The measurements are made by applying a load of 300g to heat sealed samples for 5 seconds at a pressure of 1.2 atm. and at a weld width of 2.5 cm. The actual temperature of each sample to be measured is increased by 2°C. The samples having the best seals or welds are then cut to obtain 2.5 x 10 cm. strips and the unsealed ends of the samples are hooked to a dynamometer. The S.I.T. is that temperature at which the seal or weld does not break when the 300g load is applied as described above. The S.I.T. for the propylene polymer compositions of examples 1 and 2 were of 100 and 105°C, respectively.

### Example 3

This example illustrates an air quenched blown film material comprising the propylene polymer composition and a method for preparing the same.

An air quenched blown film of the propylene polymer composition of Example 2, stabilized with 0.025 parts per hundred parts of the propylene polymer composition (pph) octadecyl-3,5-bis(1,1-dimethylethyl)-4-hydroxybenzene propanoate, 0.075 pph tetrakis[methylene(3,5-di-tert-butyl-4-hydroxyhydrocinnamate)] methane, 0.08 pph Sandostab P-EPQ composition, the main component of which is tetrakis(2,4-di-tert-butylphenyl)-4,4'-biphenylene diphosphonite and 0.05 calcium stearate and to which 0.15 pph Millad 3905 nucleator was also added, is prepared by charging the composition into a single screw extruder, extruding it through a circular die and blowing it into a film form with a sufficient amount of air to provide a film of 1 mil thickness using the following equipment and processing conditions:

Screw:	Compression ratio 3:1 to 4:1. Polyolefin barrier type L/D ratio = 24:1 to 30:1. Blow up ratio = 2.5 to 4:1. Die gap: 40 mils for a thickness of 0.5 to 5 mils.
35 Extruder barrel profile:	380 to 430°F (193 to 221°C) going from zone 1 to zone 6.
Adapter and die temperatures	: 450°F (232°C) except upper and lower die zones which are 460°F (238°C).
Screw speed:	20 rpm.
Pressure:	3000 psi (21.0 MPa).
40 The properties of the resulting film are set forth in Table 2 below.	

having a melt flow rate (MFR) of 6.0 dg/min is prepared by the cast film technique.

The properties of the resulting film are set forth in Table 3 below.

Table 3

Properties	Resins			
	Ex. 2	Ex. 2 + N	Coex <sup>1</sup>	P-E Co <sup>2</sup>
Yield Strength (MD/CD <sup>3</sup> ), psi (MPa)	2234/2121 (15.64/14.85)	3010/2837 (21.07/19.86)	2785/2635 (19.50/18.45)	2523/2452 (17.66/17.16)
Break Strength (MD/CD), psi (MPa)	6902/3387 (48.31/23.71)	6442/6317 (45.09/44.22)	5254/3520 (36.78/24.64)	4120/3363 (28.84/23.54)
Elongation at yield (MD/CD), %	16/13	16/17	10/9	15/10
Elongation at break (MD/CD), %	571/595	520/523	522/590	522/584
Elmendorf tear, (MD/CD), g/ply	44/1019	19/985	51/132	49/102
Haze	1.3	3.2	2.8	2.3
Gloss	86.7	80.4	85.0	78.1
Coefficient of friction (static/kinetic)	No slide	1.44/1.03	0.463/0.30	0.291/0.213
Moisture vapor transmission rates at 23 ° C & 90 relative humidity, g/100 sq. in/24 hrs.	0.226	0.200	0.152	0.207
As above at 37.8 ° C	0.923	0.887	0.682	0.923

<sup>1</sup> Propylene polymer composition of Ex. 2/homopolymer of propylene/propylene polymer composition of Ex. 2.

<sup>2</sup> Propylene-ethylene random copolymer with an ethylene content of 3.0%.

<sup>3</sup> MD/CD = machine direction/cross direction.

From Table 3 one can see that the propylene polymer composition of this invention provides a cast film with superior break strength, improved elongation at yield, less haze in the absence of a nucleator, improved moisture barrier properties and a better overall balance of properties.

#### Example 5

This example illustrates a biaxially oriented film material comprising the propylene polymer composition and a coextruded film of the propylene polymer composition and a homopolymer of propylene and a method for preparing same.

A biaxially oriented film of the propylene polymer composition of Example 2 stabilized as set forth in Example 3 absent the Millad 3905 nucleator is prepared by charging the composition into an extruder, extruding it through a slot die and quenching the thus formed sheet. The sheet is then oriented in the longitudinal direction between rolls running at different speeds and then passing it into a tenter frame for orientation in a lateral direction by means of diverging chains of clips. After the biaxial orientation is complete, the orientation is locked into the film by cooling. The resulting film is 1 mil thick. The following equipment and processing conditions are used:

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Extrusion profile:	Zone 1	410°F (210°C)
	Zone 2	420°F (216°C)
	Zone 3	430°F (221°C)
	Filter	430°F (221°C)
	Adapters 1 & 2	430°F (221°C)
	Die Zone 1	440°F (227°C)
	Zones 2 & 3	440°F (227°C)
	Chill Roll	86°F (30°C)
	MDO Zone 1	145°F (63°C)
	MDO Zone 8	190°F (88°C)
TDO:	Zones 1 & 2	290°F (143°C)
	Zone 3	266°F (130°C)
	Zone 4	230°F (110°C)
	Zones 5 & 6	145°F (63°C)
Screw speed:		160 rpm.
Line speed:		40 meters/min

A coextruded film 1.25 mils thick of the propylene polymer composition of Example 2 stabilized as set forth in Example 3 absent the Millad 3905 nucleator and Moplen S38F homopolymer of propylene having a MFR of 2.0 dg/min is prepared by extruding the three layer laminate and biaxially orienting as set forth above.

The properties of the resulting film are set forth in Table 4 below.

Table 4

Properties	Resins		
	Example 2	Coex <sup>1</sup>	PP <sup>2</sup>
Yield Strength (MD/CD <sup>3</sup> ), psi (MPa)	9860/5914 (69.02/41.40)	20109/6886 (140.76/48.20)	22000/10000 (154.0/70.0)
Break Strength (MD/CD), psi (MPa)	9680/5874 (67.76/41.12)	20109/5885 (140.76/41.20)	22000/10000 (154.0/70.0)
Elongation at yield (MD/CD), %	6/25	11/16	7/30
Elongation at break (MD/CD), %	6/25	11/16	7/30
Elmendorf tear, (MD/CD), g/ply	7/5	5/5	4/5
Haze	0.1	1.0	1.0
Gloss	95	90	85
Coefficient of friction (static/kinetic)	No slide	1.4/0.88	-
Moisture vapor transmission rates at 100 °F (38 °C) & 100 relative humidity, g/100 sq. in/24 hrs.	0.580	0.355	0.40
Dart impact strength, ft-lb/mil (J)	2.90 (2.23)	4.12 (3.17)	-
Shrinkage (MD/CD), %			
at 100 °C	20/32	3/3	-
at 120 °C	60/75	8/12	-

<sup>1</sup> Propylene polymer composition of Ex. 2/homopolymer of propylene/propylene polymer composition of Ex. 2, which coextruded film has a S.I.T. of 105 °C.

<sup>2</sup> Moplen S38F homopolymer of propylene having a MFR of 2.0 dg/min and a S.I.T. of about 163 °C.

From Table 4 one can see that the propylene polymer composition of this invention provides a biaxially oriented film with superior S.I.T., improved Elmendorf tear properties, less haze and superior gloss and a better overall balance of properties.

Various types of film materials of conventional thickness and thin films less than 20 mils thick to as thin as about 0.5 mils can be prepared using the propylene polymer composition described herein as well as heavy film materials, typically referred to as sheets, from 20 to 100 mils thick. For example, in addition to the films exemplified herein, it can be used to make uniaxially oriented films and extruded or calendared sheets. In addition, a layer comprising the propylene polymer composition can be applied to, e.g. by lamination or coextrusion techniques, at least one surface of a thermoplastic film material or a metallic sheet or foil substrate. Typical thermoplastic materials include crystalline homopolymers of a C<sub>2-10</sub> alpha-olefin monomer, such as propylene or ethylene, or copolymers of propylene with ethylene and/or a C<sub>4-10</sub> alpha-olefin monomer, provided that, when the alpha-olefin is ethylene, the maximum polymerized ethylene content is about 10%, preferably about 4%, and when the alpha-olefin is a C<sub>4-10</sub> olefin, the maximum polymerized content thereof is about 20%, preferably about 16%, as well as polyesters, polyamides, ethylene-vinyl alcohol copolymers and ethylene-vinyl acetate copolymers. Aluminum is a suitable metallic substrate.

In addition, film materials can be prepared from blends of from about 5 to 45% of the propylene polymer composition described herein with from about 95 to 55% of a crystalline homopolymer of a C<sub>2-10</sub> alpha-olefin monomer or copolymer of propylene with ethylene and/or a C<sub>4-10</sub> alpha-olefin monomer as described in the preceding paragraph. Preferably the amount of the propylene polymer composition present in such blends is from 10 to 30%.

The propylene polymer composition of this invention is such that one can achieve the proper balance of properties in the resulting product when a film layer of same is the material applied to at least one surface



of another thermoplastic material or to a metallic substrate and when it is blended with another thermoplastic material and the blend is used to make film materials.

The film or sheet articles prepared from blends of a propylene polymer composition with thermoplastic polymer materials can be used in packaging applications, in the production of containers for chemicals and hazardous waste and in medical applications.

The expression "consisting essentially of" as used in this specification excludes an unrecited substance at a concentration sufficient to substantially adversely affect the essential properties and characteristics of the composition of the matter being defined, while permitting the presence of one or more unrecited substances at concentrations insufficient to substantially adversely affect said essential properties and characteristics.

Other features, advantages and embodiments of the invention disclosed herein will be readily apparent to those exercising ordinary skill after reading the foregoing disclosures. In this regard, while specific embodiments of the invention have been described in considerable detail, variations and modifications of these embodiments can be effected without departing from the spirit and scope of the invention as described and claimed.

#### Claims

1. A film or sheet material comprising a blend of
  - (1) from about 5 to 45 % of a crystalline propylene polymer composition which is comprised of, by weight.
    - (i)
      - (A) from about 30 to 65 % of a copolymer consisting essentially of propylene with a C<sub>4</sub>-8 alpha-olefin containing from 80 to 98 % propylene, and
      - (B) from about 35 to 70 % of a copolymer consisting essentially of propylene with ethylene having an ethylene content of 5 to 10 %;
    - or
    - (ii)
      - (A) from about 45 to 65 % of a copolymer consisting essentially of propylene with a C<sub>4</sub>-8 alpha-olefin containing from 80 to 98 % propylene, and
      - (B) from about 35 to 55 % of a terpolymer of propylene with ethylene and a C<sub>4</sub>-8 alpha-olefin having an ethylene content of 0.5 to 5 %, a C<sub>4</sub>-8 alpha-olefin content of 1 to 9.5 %, wherein the total content of ethylene and C<sub>4</sub>-8 alpha-olefin is from 2 to 10 %;
  - and
  - (2) from about 95 to 55 % of a crystalline homopolymer of a C<sub>2</sub>-10 alpha-olefin monomer or of a copolymer of propylene with ethylene or of a copolymer of propylene with a C<sub>4</sub>-10 alpha-olefin monomer, or of a copolymer of propylene with ethylene and C<sub>4</sub>-10 alpha-olefin, provided that, when said copolymer is a copolymer with ethylene, the maximum polymerized ethylene content thereof is about 10 %, and when said copolymer is a copolymer with a C<sub>4</sub>-10 alpha-olefin, the maximum polymerized content thereof is about 20 %.

2. A film or sheet article comprising a film or sheet material as claimed in claim 1.



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㉜ **Propylene polymer films and laminates.**

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European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 95 10 3280

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.)
A	DE-A-36 24 543 (WOLFF WALSRÖDE) * page 2, line 57 - page 3, line 8 * * page 3, line 21 - page 30 *	1,2	C08L23/06 C08L23/10 C08L23/16
T	EP-A-0 483 523 (HIMONT) * page 2, line 29 - line 38 *	1,2	
T	DE-A-41 09 368 (HOECHST) * page 3, line 51 - line 54 *	1,2	
			TECHNICAL FIELDS SEARCHED (Int. Cl.)
			C08L
The present search report has been drawn up for all claims			
Place of search		Date of completion of the search	Examiner
THE HAGUE		19 July 1995	Schmidt, H
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